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IS 8883-1 (2005): Methods of sampling chemical and chemical products, Part 1: General requirements and precautions [CHD 1: Inorganic Chemicals]

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रसायन और रसायनिक उत्पादों के नमूने लेने की पद्धति

भाग 1 सामान्य अपेक्षाएँ और सावधानियाँ

(पहला पुनरीक्षण)

Indian Standard

METHODS OF SAMPLING CHEMICAL AND CHEMICAL PRODUCT

PART 1 GENERAL REQUIREMENTS AND PRECAUTIONS

(First Revision)

ICS 71.40.40, 71.100.01

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

March 2005 Price Group 8

Inorganic Chemicals and Photographic Materials Sectional Committee, CHD 1

FOREWORD

This Indian Standard (Part 1) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals and Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

This standard deals with the general requirements and precautions to be observed in sampling of chemicals and chemical products. This standard was first published in 1978. In this revision, terms relating to the statistics of sampling have been included. General precautions and principles have been elaborated further. While revision of the Part 1 of the standard, no changes have been made in Part 2 of the standard which deals with the sampling equipments for solids, liquid and gases.

There is no ISO specification for the methods of sampling chemicals and chemical products.

In this standard, considerable assistance has been derived from BS 5309 (Part 1) : 1976 (1993) 'Methods for sampling chemical products — Introduction and general principles'.

The composition of the Committee responsible for formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard
METHODS OF SAMPLING CHEMICAL AND CHEMICAL PRODUCT
PART 1 GENERAL REQUIREMENTS AND PRECAUTIONS
(First Revision)

1 SCOPE

1.1 This standard (Part 1) covers general requirements and precautions which apply in obtaining representative sample of any chemical product in the form of liquids, solids and gases.

1.2 The general instructions contained in this revised standard should not take precedence over specific sampling instructions contained in the relevant materials specifications and standards on methods of test or agreed sampling procedure between supplier and the purchaser.

1.3 Purpose of Sampling

1.3.1 *Fundamental Purpose*

Samples are taken from larger quantities or numbers in order that on examining the samples certain information pertaining the whole may be obtained. Such a procedure is necessary when the testing of the whole is impracticable. The process of sampling requires a sampling plan that will be satisfactory, if it yields the information sought, allowing for known sources of error, at an acceptable cost. Two sources of error affect the information obtained:

a) Failure of the sample or samples to represent the whole;
b) Inaccuracies in the test result:
    1) resulting from failure to apply the appropriate techniques to determine the chosen characteristics of the sample, and
    2) inherent in the test method used which may be ascertained from the precision quoted for the method.

1.3.2 *Commercial Purposes*

The fundamental purpose is relevant to all types of sampling but the commercial purposes of sampling can be conveniently divided into four groups: economic, technical, legal and safety purposes. Although safety can be said to be a technical purpose, its importance is stressed by putting it in a separate section.

1.3.2.1 *Economic purposes*

a) To serve as a basis for offer for sale.
b) To ensure that a material purchased complies with a contract specification.
c) To ensure that material for sale to customers satisfies the manufacturer's sales specification.

1.3.2.2 *Technical purpose*

a) To facilitate quality control, process control, or inspection during the process of production or manufacture.
b) To determine the nature, quantity and/or source of contaminants.
c) To establish the identity of unknown materials.
d) To confirm the identity of supposedly known materials.
e) To ensure that a given material is suitable for the purpose for which it is intended to be used.
f) To determine whether a recovered material is suitable for reuse in a process.
g) To determine the changes in the characteristics of a material with time.
h) To determine the source of the given material.

1.3.2.3 *Legal purposes*

a) To ensure that material satisfies statutory requirements.
b) To ensure that effluents from a processing site conform to permitted composition limits.
c) To provide material for forensic investigations.
d) To assess material for statutory liability.
e) To assess material for arbitration purposes.

1.3.2.4 *Safety purposes*

a) To establish whether a given physical situation is safe or hazardous.
b) To help establish the cause of an accident.
c) To classify the hazards of the material.

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
4905 : 1968	Methods for random sampling
8883	Methods of sampling of chemicals and chemical products:
(Part 2/Sec 1) : 1978	Sampling equipment, Section 1 For solid
(Part 2/Sec 2) : 1978	Sampling equipment, Section 2 For liquid
(Part 2/Sec 3) : 1978	Sampling equipment, Section 3 For gases

3 TERMINOLOGY

For the purpose of this standard the following definitions shall apply:

3.1 Consignment — A quantity of material covered by a particular consignment note or shipment document.

3.2 Lot — The total material to be sampled using a particular sampling plan. It may consists of a number of consignments, batches or items.

3.3 Batch — A definite quantity of material that may be one item or a number of items which belong together because of their manufacture or production under conditions which are presumed to be uniform.

3.4 Sub-items — The practical or hypothetical division of an item in connection with the assessment of variability.

3.5 Increment — A quantity of material taken from the lot, in a single operation of the sampling equipment.

3.6 Test Portion — The portion withdrawn or taken from the test sample and used in the test or observation.

3.7 Sample — One or more portions of material derived from a larger amount in one item, or one or more items taken from a larger number of items. Alternatively one or more items taken from a population and intended to provide information on the population and possibly to serve as a basis for a decision on the population or on the process which had produced it.

NOTE — The sampling of bulk materials, of discrete population of continuous materials, such as sheet metal, requires the use of terms specific to their nature. In the sampling of solid bulk materials (powders, coal, coke etc), the word 'increments', 'gross sample', 'laboratory sample' and 'test sample' are used while the word 'specimen' is used in connection with the sampling of discrete population.

3.8 Sampling Plan — The planned procedure of allocation, withdrawal and preparation of a sample or samples from a lot to yield the required knowledge of the characteristic(s) from the final lot sample with a minimum cost, effort or delay appropriate to an acceptable sampling error so that a decision can be made regarding the lot.

3.9 Sample Preparation — For bulk materials, the set of operations, such as reduction of size, mixing, dividing, etc, necessary to transform a bulk sample into a laboratory or test sample.

3.10 Gross Sample — A sample as collected from lot by mixing the increments.

3.11 Reduced Sample — A sample that has been obtained by reducing the quantity of another sample without change of composition.

3.12 Composite Sample — A blended bulk sample, the individual samples of which have been taken in amounts proportional to the quantity of the material that they represent.

3.13 Sub-sample — A smaller sample produced in a specified manner by the reduction in volume or quantity of the gross sample.

3.14 Representative Sample — A sample assumed to have the same composition as the material sampled when this is considered as a homogeneous whole.

3.15 Laboratory Sample — A sample, intended for inspection or testing, prepared from the final lot sample or otherwise obtained.

3.16 Test Sample — The sample prepared from the laboratory sample from which the test portions are withdrawn or taken.

3.17 Reference Sample — A sample, prepared from the final lot sample at the same time as and identical with the laboratory sample, accepted by the parties to a contract and which is held for use as a laboratory sample in the case of dispute.

3.18 Storage Sample — A sample, prepared from the final lot sample at the same time and identical with the laboratory sample, intended for possible future use as a laboratory sample.

3.19 Test or Observation — An operation made in order to measure or classify a characteristic.

3.20 Characteristic — A definite property of material. It will differentiate either qualitatively (by attributes) or quantitatively (by variables).

3.21 Automatic Sampler — A device which, when correctly installed and when activated by appropriate control equipment, enables a representative sample of flowing material to be taken automatically.

3.22 Self-Actuated Sampler — An automatic sampler that is operated by material flow or pressure.

3.23 Reduction

a) The process which reduces the quantity of the sample.
b) The process which reduces the particle size of the material constituting the sample.

3.24 Ullage (Outage) — The capacity of a container not occupied by the material, or the distance between the surface of the material and some fixed reference point at the top of the container.

NOTE — In the case of liquids this allows for its expansion.

3.25 Terminology Peculiar to Solids

3.25.1 *Sampling Unit* — It may be defined as that portion of the material which is chosen in such a manner that there is a high probability that it will represent the entire bulk of the material.

3.25.2 *Fluidized Solids* — A solid reduced to the powder form so that it can be piped or made to flow like a liquid, for example cement.

3.25.3 *Unit Sample* — The quantity of material collected at a particular point of time from the conveyor.

3.26 Terminology Peculiar to Liquids

3.26.1 *Average Sample* — One that consists of proportionate parts from all sections of the container.

3.26.2 *All-Levels Sample* — One obtained by submerging a closed sampling instrument to a point as near as possible to the draw-off level, then opening the sampling instrument and raising it at a rate such that it is about three-fourth full as it emerges from the liquid. An all-levels sample is not necessarily an average sample because only in the case of tankers with uniform cross-section, an all-levels sample and average sample will be same. In most of the instances the operator may not be able to raise the sampling instrument at the variable rate required for proportionate filling.

3.26.3 *Upper Sample* — A spot sample taken in a liquid at a level of one-sixth of the volume of the liquid, below its surface.

3.26.4 *Middle Sample* — A spot sample taken in a liquid at a level of one-half of the volume of the liquid, below its surface.

3.26.5 *Lower Sample* — A spot sample taken in a liquid at a level of five-sixth of the volume of the liquid, below its surface.

NOTE — The height of the container should be divided into six parts as 1/6, 2/6, 3/6, 4/6, 5/6 and 1 calibrated from the bottom of the container.

3.26.6 *Single Tank Composite Sample* — A blend of the upper, middle and lower sample. For a tank of uniform crosssection, such as an upright cylindrical tank, the blend consists of equal parts of the three samples. For a horizontal tank, the blend consists of the three samples in the proportions.

3.26.7 *Top Sample* — A sample taken not more than 15 cm below the top surface of the liquid.

3.26.8 *Outlet Sample* — One normally obtained at the level of the tank outlet. In the case of swing pipe outlet, the height of the pipe inlet end shall be noted.

3.26.9 *Continuous Sample* — A sample taken continuously from a stream so as to obtained a representative sample.

3.26.10 *Mixed Sample* — One obtained after mixing or vigorously stirring the contents of the original container and then pouring out or drawing off the quantity desired.

3.27 Terms Peculiar to Cases

3.27.1 *Blended Bulk Sample* — A collected set of sample blended together to obtain a uniform sample.

3.27.2 *Spot-Sample* — A sample of specified number of size taken from a specified place in the material or at a specified place and time in a stream and representative of its own immediate or local environment.

3.28 Terms Relating to the Statistics of Sampling

3.28.1 *True Value* — The unequivocal value of a characteristic of the material. The true value is an ideal notion and in general it cannot be known.

3.28.2 *Average Lot Quality* — The result that would theoretically be obtained by taking the average of results from a large number of laboratories of replicate determinations of a characteristic, by the test method in question, made on replicate samples taken from the lot.

3.28.3 *Variability* — The differences throughout the material in the value of a characteristic.

3.28.4 *Homogeneous Material* — Material in which the mean values of the characteristic(s) under consideration of the component portions are not significantly different.

3.28.5 *Heterogeneous Material* — Material in which the mean values of the characteristic(s) under consideration of the component portions show significant variation.

3.28.6 *Defect* — A failure of an item to meet a specified requirement for the value of a characteristic.

3.28.7 *Defective Item* — An item containing one or more defects.

3.28.8 *Error* — A discrepancy from what is true or right.

3.28.9 *Accuracy* — The closeness of test results to the true value.

3.28.10 *Precision* — The closeness of agreement between the results obtained for the same characteristic of identical test material, by applying the test procedure several times.

3.28.11 *Sampling Error* — The error arising from known and acceptable deficiencies in the sampling plan.

4 GENERAL PRECAUTIONS AND PRINCIPLES

4.0 In drawing, storing, preparing and handling test samples, following general precautions shall be observed.

4.1 Sample shall not be taken at a place exposed to such weather as would affect basic change in its physico-chemical characteristics.

4.2 Sampling shall be done in the presence of purchaser and the supplier or their representatives or as per the agreed sampling plan. Each sample container after being properly filled and closed shall carry the seal and signatures of both the parties or their representatives or as agreed. The sample container shall be properly labelled with necessary information such as:

a) Name of the sample;
b) Source of sample;
c) Date/Time of sample;
d) Drawn by;
e) In the presence of; and
f) Special precautions, if any.

4.3 Use of appropriate personal protective equipment (PPE) are strongly recommended during sampling.

4.4 Never smell the sample or taste by tongue.

4.5 Adequate training of sampling is must.

4.6 Proper sampling tools/containers need to be used.

4.7 Adhere to the local regulatory guidelines, if any during the sampling.

4.8 The sample container shall be of such a size that they almost but not, completely filled by the sample. They shall be made of the material which shall not react with the contents of the sample. In other words the sample shall not change its. physicochemical characteristics in the container used.

4.9 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means or appropriate procedures.

4.10 Precautions shall be taken to protect the samples, the sampling instrument and the container of the sample from adventitious contaminations.

4.11 Once the container is opened there is a risk of deterioration of contents due to the presence of various factors, such as air, light, moisture, excessive temperature, etc. Care has to be taken to avoid the risk of polymerization, formation of peroxides in the sample by undue exposure to air or strong light.

4.12 To obtain samples that are representative in the statistical sense, one must consider such factors as physical form, uniformity, type and number of containers. All these factors influence the choice of method for performing the sampling operation, as well as the number and storage of the required samples.

4.12.1 Time interval shall be evenly fixed through the entire time of flow of the material for an ideal gross sample.

4.13 Uniformity of Sampling

During unloading, re-loading or loading the product which proceeds at a uniform rate or when a constant amount of product loaded during a unit of time, unit samples and increments of the unpacked product should be taken in equal quantities at uniform time intervals.

4.13.1 In case, samples are taken regularly, uniformity with respect to time should be observed.

4.13.2 In loading, unloading and re-loading the product at a non-uniform rate of speed-packages and increments of even sizes should be taken at time intervals determined by the rate of flow of the product and the quantity of sample required. Alternatively increments of different sizes may be taken at different time intervals, taking into account the rate of flow of the product and quantity of the sample desired.

4.14 Mixing Packed Liquids, Semi-liquids, Greasy and Pasty Products

Before taking the increments (if not stated otherwise in the standard for the given product), the product in the selected unit packages should be thoroughly mixed with the use, if possible, of mechanical means. If the product is permanently delaminated — sample should be taken from each layer separately, in proportion to the mass of the product in the individual layer.

4.15 Taking Increments

4.15.1 *Packed Products*

Increments should be taken from unit packages selected from the consignment at random. Each increment should be taken from a different place in the package. The places of taking the increments should be spaced as evenly as possible inside the package. Efforts should be made to take increments from the whole thickness of the layer of the product.

4.15.2 *Unpacked Products*

4.15.2.1 *Liquid products*

Increments should be taken while the product is being pumped over. In ~~the case~~ of turbulent flow or if the liquid is mixed over, a side cock should be placed in the pipe through which the liquid is flowing and the increments should be taken from the cock.

If for justifiable reasons it is not possible to take the increments during the process of pumping the product over, and the reservoir is provided with hatches, a testing tube should be used for sampling.

The use of the testing tube depends on the depth of the reservoir and the height of the column of the liquid. In cases where a testing tube cannot be applied, it is permissible to use other increments.

Increments should be taken over the whole height of the column of the liquid, from places spaced as evenly as possible inside the reservoir.

4.15.2.2 *Semi-liquids, greasy, pasty, loose and lumped products*

Each increment should be taken from a different place in the product. The places from which the increments are taken should be spaced as evenly as possible, in the mass of the product. Increments should be taken over the whole thickness of the layer of the product.

4.16 Following precautions about time and place of sampling for liquids, liquefied gases and fluidized solids shall be observed.

4.16.1 *Finished Products*

When loading or discharging finished products, samples should be taken from both shipping and receiving tanks, and from the pipeline if required.

4.16.2 *Ship or Barge Tanks*

Each product should be sampled immediately after the vessel is loaded or just before discharging.

4.16.3 *Tank Cars*

Sample the product immediately after the car is loaded or just before unloading (after ensuring sufficient homogeneity of the product).

5 PRINCIPLES AND PRECAUTIONS PECULIAR TO SOLIDS

5.1 Every sample should be collected and prepared in strict accordance with specified procedure.

5.2 Because of many variations in the conditions under which solids must be sampled and in the nature of the material being sampled, it is essential that the samples be collected by a trained and experienced sampler. Because of the variations in the manner of handling the solid, it is impossible to specify rigid rules describing the exact manner of sample collection. Correct sampling principles must be applied to conditions as they are encountered.

5.3 To be able to make probability or confidence statements about the property of a lot, the sampling procedure must allow for some element of randomness in selection because of the possible variations in the quality of the material. Generally, where segregation is known to exist, and random variation of quality is not possible, the sampling should be designed to allow for this. The sampler should always be on the alert for possible biases arising from the use of a particular sampling device or from unexpected segregation in the material. Generally, where sampling is to be applied to the output of a given process on a continuous basis, it will be desirable before adopting a particular sampling plan, to undertake an extensive preliminary study of variation in the material and possible biases in the sampling instruments and methods of reduction.

5.4 Whenever possible, non-package, bulk materials should be sampled while the material is in motion rather than in static piles, carloads, etc. Such occasions are frequently ideal for the application of falling-stream samplers.

5.5 Sampling of bulk solids from boxcars, barges, etc, introduces additional problems because of possible non-uniformity in particle size, moisture, impurities, etc. The statistical treatment is complex and beyond the scope of this recommended practice.

5.6 All auger methods and all scoop methods used on materials not being loaded or transferred fail in prime sampling requirement that of random selection of the particles or portions selected as samples. Scoops and shovels are limited to use at or near the top surface. Augers and thieves are normally inserted in a pre-set pattern. Consequently, particles on the bottoms or along certain sides of containers never have an opportunity to be included in a sample. For heterogeneous or valuable material, this alone may furnish sufficient reasons to go to a falling-stream sampler.

5.7 Because of the above factors, the recommended procedures that follow are limited to the mechanical operations of taking the required number of increments called for in a purchase contract.

5.8 The sampling equipment, sample preparation equipment, containers, etc, used for sampling must be clean, dry, uncontaminated and inert to the material being sampled and protection from heat, cold, light, loss or gain of moisture may be necessary.

5.9 Analytical reagent grade chemicals generally are packed in containers marked with full details of analysis. It is recommended that sample containers, as far as possible, be marked with full details necessary for such chemicals. The labels on the bottles shall be examined for the completeness of details before opening and taking out samples.

5.10 Collection of Gross Sample

When the shipment is large and frequent section variations do not prevail, and it is being transferred by containers with a capacity approximately equal to the sampling unit, it is advisable to increments for the gross sample. When the accumulated gross sample is too large to take the entire content of a definite percentage of the containers or increments for the gross sample. When the accumulated gross sample is too large to handle conveniently as a unit, it is permissible to reduce the gross sample in small sections by the specified procedure.

5.10.1 *From Truckload Lots*

When the material is to be unloaded from flat bottomed trucks or cars, the top surface of the truck shall be divided into eight equal areas and from each of these areas the material equivalent to one sampling unit shall be taken in the following manner. From a suitable bank of the material, beginning from the bottom, take with a shovel or other suitable instrument at regular and equal intervals from bottom to the top of the bank, ten equal portions of the material as specified. Combine these sampling units for the gross sample. When the accumulated gross sample is too large to handle conveniently as a unit, it is permissible to reduce the gross sample in small sections by the specified procedure and subsequently combine the reduced portions in the correct ratio.

5.10.1.1 When the material is being transferred from to the truck by means of wagons, take the specified increments according to the material in question from specified number of conveying containers. Combine these increments to form the gross sample.

5.10.2 While sampling from barrels, bags, etc, take the specified increments from a specified number of containers and combine them for the gross sample.

5.10.3 While sampling from hoppers, storage piles, etc, daily or periodic sample may be taken. For this purpose specified increments may be taken from the lot and combined for the gross sample.

5.10.4 When the material is a fine powder, the sampling unit is taken by means of a suitably designed thief or shovel [*see* IS 8883 (Part 2/Sec 1)].

5.10.5 When the material is thoroughly wet but not dripping, sample may be taken by any of the sampling implements discussed in **5.10.1** to **5.10.4**. When the solid material is uniformly suspended in liquid, several vertical sections of the material may be taken by means of a suitably designed sampling implement and combined for the gross sample.

5.10.6 When hygroscopic material is contained in airtight containers which may be readily opened and closed, take a sample by means of suitably designed thief. When such material can be sampled while being transferred to the final container for shipment, small and equal increments at uniform and regular intervals are taken directly from the stream by means of suitable dipper and combined for the gross sample.

5.11 Preparation and Reduction of Sample for Solids

5.11.1 *Appearance*

Visual inspection of the sample is recommended to determine if the material contains gross contamination, or if it is equal to the approved sample. It may show if the material has picked up excessive moisture, or if further laboratory processing is required to reduce the material to a more uniform particle size. Further testing should cease in case of unusual appearance until another sample is obtained, and the cause for the abnormality has been established.

5.11.2 *Screening*

If extraneous matter is detected, a decision shall be made as to whether it belongs to the sample. Dirt or other contamination may actually be in the lot and properly belong in its proportionate part in the sample. A careful consideration of each individual case shall be made, to determine if the contaminant should be removed by passing the sample through an appropriate screen.

5.11.3 *Grinding*

Coarse or non-uniform samples may require grinding in a mortar, a mill, or other suitable devices to obtain a more uniform sample. The sample may be subjected to grinding, or it may be more efficient to screen off the oversize, grind it and then blend all portions together.

5.11.4 *Minimum Sample Size*

Where analysis of a composite sample is specified or permitted, individual sample increments are combined to form the gross sample which is reduced to make it suitable for laboratory handling or analysis. The minimum permissible quantity of a laboratory sample is the quantity sufficient to carry out all the tests for a single analysis.

5.11.5 *Sample Preparation Scheme*

In general, a sample preparation scheme shall consist of particle size reduction, blending, splitting and a repeat of this series of operations until the desired minimum sample size is attained. It is difficult to write a general scheme for the reduction of a sample for all types of material because of the nature of the material and the purpose of sample. It is important, however, that any splitting operation be immediately preceded by blending. Two standard operations for reduction are given by the following procedures.

5.11.5.1 *Cone and quarter method*

Dump the individual increments on to a clean canvas, and shovel into a pile, placing each shovelful on the top of the pile. Flatten the apex of the cone with a shovel or a board until it is about one-fourth its original height. Divide the pile into four equal parts by drawing a board twice through the centre of the pile, making right-angle cuts. Discard the opposite quarters, chosen at random and combine the remaining quarters into a cone-shaped pile. Repeat the above operation until the desired quantity of sample is obtained. Place the final sample on a clean canvas and mix by alternately raising opposite corners of the canvas.

5.11.5.2 *Sample splitter or riffle*

The sample splitter or riffle should be constructed of a material suitable for use with material under test. It consists of a series of chutes that are directed alternately to opposite sides. The slot width should be at least three times the diameter of the largest particle to be passed through to prevent bridging and therefore biased splitting. Pass the composite or gross sample through the riffle to divide it into two approximately equal portions. Pass one of these portions, selected at random again through the riffle. Continue the operation until the sample size is reduced to either that required or the minimum sample size beyond which additional grinding is necessary.

5.11.6 *Blending*

Sample homogeneity must be assured by thorough blending prior to the analysis. The operation should be performed on all samples in such a way that they will not be changed because of light sensitivity, hygroscopicity, etc. No sample container should be filled more than approximately half-full and no container should be opened for sample removal until it has been tumbled on a mechanical blender designed for the purpose or rotated by hand, end over end, at least 25 revolutions. On any sample, blending should be done after screening or grinding or both.

5.12 While sampling semi-solids, greasy and pasty products, sampling equipment, size of the increment quantity of sample required will be different from the corresponding ones for solids, powders and pellets.

6 PRINCIPLES AND PRECAUTIONS PECULIAR TO LIQUIDS

In general, a uniform and representative sample of liquid material is a feasible possibility because of the usual absence of segregated constituents, the ease with which the separate components may be intimately mixed and the degree of comminution to which a liquid may be subjected without producing a separation of the individual components.

6.1 When the liquid is thin, non-viscous, and does not contain immiscible constituents, a homogeneous condition usually exists and a sample from any part of the bulk is sufficiently representative of the whole. When viscous or immiscible materials are present, a heterogeneous condition usually exists and extreme care must be exercised to obtain a thoroughly representative sample. The proper selection of a representative sample, therefore, involves a consideration of the physical laws of liquids, their chemical activity, the miscibility of all components, and the interference of any insoluble materials carried by the liquid.

6.1.1 *Sampling Liquids in Quiescent State*

All liquids not in motion may be sampled by obtaining portions at specified points from the top to the bottom of the liquid by means of a suitable sampling apparatus.

6.1.2 *Sampling Liquids in Motion*

6.1.2.1 *From a pipe or conduit carrying a varying fraction or flow under a constant or varying head*

A continuous or intermittent sample may be taken by inserting a small pipe into the line on the discharge side of the pump or propelling force of the flow. The sampling tube should extend to one-half of the distance to the centre of the flow, with the inner open end turned at an angle of 90° and facing the flow of the liquid. The sampling tube may be supplied with a stopcock in order to regulate the flow into a receiver. The amount drawn during the period of taking a continuous sample constitutes the gross sample.

6.1.2.2 *From a pipe or conduit carrying a varying fraction or flow*

By means of the appropriate sampling instrument a definite fraction of varying flow can be collected over a required period of time constituting the gross sample.

6.1.2.3 *Liquids containing insoluble matter in suspension*

a) When the sediment remains a reasonably uniform suspension during the period required for taking the sample obtained by appropriate sampling instrument, the liquid is thoroughly mixed and the sample obtained by one of the aforesaid procedures.

b) When the sediment settles rapidly or is impossible of uniform distribution, it is necessary to take special precautions to secure the components of the sample in the proper proportion. This may be accomplished with a fair degree of accuracy by mixing the liquids thoroughly and then rapidly taking a complete column of the liquid by means of suitable thief.

c) To accurately sample a liquid containing insoluble matter it is necessary to filter the entire liquid, weigh the insoluble material and filtrate separately and then take separate samples of the sediment and liquid recording the ratio of sediment to liquid for use in the final evaluations of the original material. This, however, is impracticable except in such instances wherein the size of sample allows of economical handling in the manner described.

6.1.2.4 *Liquids composed of immiscible layers*

The most practical and accurate procedure in this case is to effect a separation of layers and combine portions of each layer in the proper ratios.

6.1.2.5 *Liquids containing crystals due to supersaturation*

Frequently conditions are such that a portion of the liquid or certain constituents thereof, have crystallized in the container. Oleum and acetic acid are common examples of this class. When practical, the material should be warmed sufficiently to dissolve completely or melt the crystals before sampling. Otherwise if the crystals are small and remain in suspension the mass should be sampled as a liquid containing insoluble matter in suspension.

6.1.2.6 *Volatile liquids*

Such liquids are best sampled from a continuous flow while the liquid is being discharged or by means of a siphon from containers. If the liquid is uniform in composition or can be made so, a portion may be run to the bottom of the sampling bottle until the bottle overflows through another tube from the top into another bottle or to the waste pipe, to such an extent as to leave a liquid in the first bottle containing maximum amount of the volatile constituent. The sample bottle should then be quickly stoppered and sealed for transportation to the laboratory.

6.1.3 *Bulk and Small Containers*

6.1.3.1 *Bulk containers*

In the case of wagons, drums, tanks, etc, each container shall be sampled. While taking samples from bulk containers, increments may be taken at suitable points with a random start from wagons and drums.

6.1.3.2 *Small containers*

These containers shall be sampled at random. In order to ensure the randomness of selection, procedures given in IS 4905 may be followed. Simple random selections or systematic selections with a random start are generally recommended.

6.1.3.3 For important characteristics given in the standard, individual samples shall be tested. The remaining characteristics in the specification shall be tested on a composite sample.

6.1.3.4 For tanks and large containers an all-levels sample shall be taken for example in the case of petroleum and diesel products. Single composite sample shall be taken when the composition of contents is not homogeneous, for example in the case of paints.

6.2 Number and Location of Samples

6.2.1 *Bulk Containers (Tanks, Tankcars, etc)*

If the liquids in bulk containers are found to be homogeneous only limited sampling shall be usually required. Upper, middle and lower samples or top and outlet samples may be individually tested to confirm this, by means of simple physical tests, such as refractive index, specific gravity, viscosity, etc. Complete testing may then be performed on a composite sample.

6.2.2 *Packed Materials (Drums, Cans, Bottle, etc)*

In the ease of lots of drums, bottles and cans, the homogeneity of the lot may not be assumed, and the required number of samples should be determined in accordance with general sampling and statistical considerations. The specific containers to be sampled for individual testing should be chosen by means of a table of random numbers (*see* IS 4905).

6.3 Sampling Operation

6.3.1 Procedures for sampling cannot be made explicit enough to cover all cases. Extreme care and good judgement are necessary to ensure samples which represent the general character and average condition of the material. Clean hands are important. Clean gloves may be worn only when absolutely necessary, such as during cold weather, or for reasons of safety. Wiping cloths should be selected so that lint is not introduced, contaminating samples.

6.3.2 *Corrosive Liquids*

Many liquids evolve vapours which are toxic and flammable. Avoid breathing such vapours and provide adequate ventilation. Avoid ignition from open flames or sparks produced by static electricity or by metal equipment or tools. Do not wear hobnailed shoes. For instance anhydrous hydrogen fluoride is a highly corrosive liquid which boils at 19.5°C. It attacks glass, has a great affinity for water, and its vapour is irritant and toxic. Its action on the skin and eyes is strongly corrosive, producing severe and painful burns which may not be immediately evident and which respond slowly to treatment. In such a case samples should be handled only inside a well-ventilated fume cupboard. Rubber gloves, boots and gown of a suitable size to give adequate protection to individual, and full head and face protection shall be worn when handling the material. In the event of contact or suspected contact, flood with water and seek immediate medical attention.

6.3.3 *Volatile Liquids*

When sampling relatively volatile products, the sampling apparatus shall be filled and allowed to drain before drawing the sample. If the sample is to be transferred to another container, this container shall also be rinsed with some of the volatile product and then drained. When the actual sample is emptied into this container, the sampling apparatus should be opened into the opening of the sample container and remain in this position until the contents have been transferred so that no unsaturated air will be entrained in the transfer of the sample.

6.3.4 *Non-volatile Liquids*

When sampling non-volatile liquid products, the sampling apparatus shall be filled and allowed to drain while drawing the actual sample. If the actual sample is to be transferred to another container, the sample container shall be rinsed with some of the product to be sampled and drained before it is filled with the actual sample.

6.3.4.1 Samples shall be taken in a protected area and kept away from flames.

6.3.5 A sample shall be considered suspect under any of the following circumstances and should be referred to the appropriate supervisor before analysis:

a) Sample container is damaged or defective;
b) There is any doubt as to the nature of the contents of the sample container, for example, because of the presence of an old label, incorrect marking or insufficient identification;
c) There is evidence of an unexpected lack of uniformity, for example, a separate layer or suspended matter;
d) Obvious and unusual variations are apparent in the sample;
e) The container closure is loose, whether or not there is evidence of leakage; and
f) Evidence that the closure or liner has been attacked.

6.4 Size of the Sample

The quantity of the sample should be specified by the test instructions or at least three times greater than the minimum necessary for the actual tests.

6.5 Precautions Before Shipping

6.5.1 *Volatile Samples*

It is necessary to protect volatile samples from evaporation. Transfer the product from the sampling apparatus to the sample container immediately. Keep the container closed except when material is being transferred.

6.5.2 *Light-Sensitive Sample*

It is important that samples sensitive to light be kept in the dark if testing is to include the determination of such properties as colour, inhibitor content, stability test, or neutralization values. Brown glass bottles may be used. Wrap or cover clear glass bottles immediately. It is a definite advantage to use covered metal or cardboard containers into which the sample bottles may be placed immediately after collection.

6.5.3 *Materials of High Purity*

Protect highly refined products from moisture and dust by placing paper, plastic, or metal foil over the closure and the top of the container.

6.5.4 *Container Outage*

Never completely fill a sample container, but allow adequate room for expansion, taking into consideration the temperature of the liquid at the time of filling and the probable maximum temperature to which the filled container may be subjected.

6.5.5 It is of prime importance that the person in charge of sampling be aware of the final purpose of the sample in the laboratory, so that precautions are not carried too far, since they may be superfluous in procuring a sample which is sufficiently representative for the required determinations. On the other hand, one should not overlook details which are important if the operation is to be successful and accurate results are required. If same requirements are to be omitted because they are considered superfluous for the sampling operation, this should be recorded in the official document drawn up and if at all possible, on the label of the bottle sent to the laboratory.

6.6 Shipping Precautions

To prevent the loss of liquid during shipment and to protect against moisture and dust cover, the closure of the glass bottle with plastic caps which have been swelled in water, wiped dry, placed over the top of the stoppered bottle and allowed to shrink tightly in place. Screw-top bottles are recommended. The cap should be lined with material inert to the sample. The screw caps should be secured by the use of adhesive tape or similar materials.

NOTE — Certain industrial chemicals are limited as to size, packaging, and method of transporting. The shipment of samples of flammable liquids is subject to the relevant national and local regulations.

7 PRINCIPLES AND PRECAUTIONS PECULIAR TO GASES

7.0 General

7.0.1 The proper sampling of gases often presents greater difficulties than the analysis. This is particularly true when it is desirable to obtain a representative sample of a gas during a considerable period of time as it passes through a pipe or flue from the source of production. Gases travel through straight pipes and conduits in an irregular succession of waves with a spiral motion; the maximum velocity is altered in a varying degree by every bend and obstruction throughout the time of travel. Where gases are encountered which are subject to temperature changes either above or below normal this factor shall also considerably interfere with proper sampling. The temperature of gases will vary throughout the cross-section of the pipe, usually being hottest at points of maximum velocity and coldest next to the walls and in dead pockets.

7.0.2 Gases are to be usually sampled from a flow possessing varying velocity, temperature and composition. Under such conditions it becomes necessary to collect a sample over an extended period whereby the number of series of gas waves sampled is such that there is a high probability that a true and representative sample will be obtained. Under some conditions, useful and more definite data regarding composition and gas-flow is obtained by taking frequent 'grab' samples. It is, therefore, quite evident that the subject of gas sampling naturally divides itself into two phases for consideration — that of continuous sampling and 'grab' or instantaneous sampling.

7.0.3 For drawing samples from gases flowing through pipes, appropriate sampling tube should be used. This tube should be inserted so that the longest tube reaches the centre of the flowing gas.

7.0.4 In order that the sample should be truly representative of the total gas flowing during a definite period, it is necessary to constantly draw a definite proportion of the gas.

7.0.5 While sampling gas, special care shall be taken about the sampling equipment while taking the 'grab' or a continuous sample [*see* IS 8883 (Part 2/Sec 2)].

7.0.6 *Sampling Plan*

When planning sampling operations for gases it is necessary to consider the following points:

a) Nature of the information to be derived from the programme of sampling and analysis; and
b) Whether the gas will be static or in flow, and the prevailing conditions of pressure and temperature.

7.0.7 *Purposes of Sampling*

The type of sample taken, and the technique used, may depend upon the purpose of the exercise. The most common reasons for examining a sample are:

a) to determine whether the gas complies with a specification;
b) to assist in the control of manufacturing process;
c) to indicate the changes of composition occurring during manufacture over a period of time;
d) to detect hazardous conditions which may arise, for example, due to failure of purification process;
e) to determine the nature or quantity of an abnormal impurity; and
f) to determine whether an omission conforms to permitted limits of impurity.

When sampling a solid or liquid product, it is usual to consider a convenient basic unit of material, such as a batch or a consignment to be examined. This concept does not always apply to gas sampling because gas production, and sometimes gas distribution, is often a continuous process. The gas may reach the consignee through a pipeline within a short time of manufacture, and any variation of quality is considered in terms of time and flowrate rather than to a finite quantity of gas required to be sampled. In practical terms, a consignment can relate only to gas supplied in cylinders, each cylinder or a group of related cylinders forming a sampling unit.

7.1 Types of Sample

In practice, the sample required is usually a spot sample, a blended bulk sample, a weighted sample, an intermittent or a continuous sample. Under suitable circumstances any one of these types may be representative either of a finite quantity of gas within a container, or of a quantity of gas passing through a certain section of a pipeline during a stated period of time at a stated pressure.

7.2 Sampling to Suit the Analytical Requirements

The modern development of instruments for gas analysis has influenced sampling practice. Following the widespread adoption of gas chromatography, infrared analysis, and other physics methods, it is now common practice to monitor product quality continuously, and often automatically. A sampling line is installed to supply gas to the analyser rather than to fill a sample container which is later taken to a laboratory.

7.2.1 When a sample within a container is required, the volume of gas taken must be sufficient for the analysis specified; this volume may vary widely according to the methods employed. The analyst may require a certain type of container, or a minimum sample pressure suitable for the tests planned, and may need more than one sample to complete his programme.

7.3 Pre-concentration and Purification Techniques

When it is necessary to determine a constituent present at very low concentration, the sampling may be combined with a preliminary stage of the analysis. A metered volume of gas is passed through a trap containing a suitable absorbent or reagent for the concentrate. A constituent which is known to interfere in a method of analysis may be removed or rendered harmless during the sampling by placing a suitable reagent vessel upstream of the sample container.

7.4 Factors Influencing the Method of Sampling

7.4.1 Although it is commonly assumed that a given quantity of gas quickly reaches a uniform composition by diffusion, this is not invariably the case, and it is necessary to consider the possible exceptional circumstances.

7.4.2 *Compressed Gas in Cylinders*

The contents of each cylinder are homogeneous and sampling presents no problem, subject to the following:

a) It is customary to specify a minimum pressure in the cylinder when sampling a permanent gas which may contain as an impurity a vapour notably moisture. The measured moisture content rises as the cylinder approaches depletion.

b) When a gas mixture has been prepared by introducing the components successively into a cylinder at the appropriate partial pressures, a substantial period of time may elapse before diffusion is complete, particularly when the components differ widely in density.

7.4.3 *Sampling from Gas Holders*

When the contents of gas holder are replenished stratification may persist for a considerable time. It is hardly practicable to obtain a representative sample from a larger holder, and the usual practice is to sample gas passing into service from the pipeline leaving the holder.

7.4.4 *Sampling from a Pipeline*

The composition of the stream may be variable in space or in time for the following reasons.

7.4.4.1 *Imperfect mixing of constituents*

Stratification is most frequently encountered when the pipe has a large diameter and gas flow is slow. It is important to select the optimum location of the sampling point; taking advantage of the mixing induced by any turbulent which may occur down-stream of a bend, a change of cross-sectional area or an obstruction in the pipe. Since there is less possibility of stratification persisting in a rapid stream, it is an advantage to sample where the pipe has minimum internal diameter. Avoid sampling at any point where the gas is stagnant, for example, immediately downstream of an orifice plate or other sharp edged obstruction. The extent of stratification occurring at any selected point may be investigated by using a mobile probe to collect a number of samples in rapid succession across the section of the pipe. Analysis of these samples will indicate the optimum position of the probe to obtain a representative sample.

7.4.4.2 *Presence of a non-gaseous phase*

The presence of entrained solids or liquid droplets, which require to be included quantitatively, may necessitate sampling isokinetically.

7.4.4.3 *Cyclic or periodic fluctuation of composition*

The sampler may proceed by one of the following alternative methods, depending upon the information required from the analysis:

a) To take a series of spot samples at appropriate time intervals. The analysis will indicate the extent of fluctuation related to time.

b) If both the gas composition and the flow rate are variable, an average composition may be derived by sampling at a constant rate from a number of equally spaced points across the section of pipe.

   After analysis each sample the average composition ($^{-}$) is calculated from the formula:

$$\bar{V} = \text{sampling} \ \frac{V_1x_1 + V_2x_2 + V_3x_3 + \ldots}{V_1 + V_2 + V_3 + \ldots}$$

   where V_1, V_2, V_3, etc, are proportional to the gas velocities at the several points and x_1, x_2,

x_3, etc, are the corresponding analyses of the samples taken at these points.

c) To obtain a representative multi-point sample space several sampling points at equal distances across the section of the pipe and adjust the rate of collection at each point to bear a constant ratio to the gas velocity at that point, using an apparatus designed to blend the sub-sample stream continuously.

d) Continuous sampling into a recording analyser will indicate the range of variation of composition, and average value can be calculated.

8 LABORATORY SAMPLE AND STORAGE PRECAUTIONS

8.1 Method of Preparing a Laboratory Sample

8.1.1 *Liquid, Semi-liquid, Greasy and Pasty Products*

The increments taken should be placed in a clean, dry container of suitable size, such as a barrel, a stoneware vessel, or a bottle. The gross sample obtained in this way should be thoroughly mixed and then a laboratory sample should be taken from it.

8.1.2 *Loose and Lumped Products*

The increments taken should be heaped in a clean place, protected from contamination of the product lines. If the product *is* in lumps, they should be hand-crushed or machine-crushed into particles below 25 mm. A sample prepared for the investigation of the granulometric content should be made of a material which is harder than the broken up product and which does not introduce into the sample any impurities liable to change the results of the testing.

8.1.2.1 In the case of distinctly foreign particles being present in the sample such as hits of foreign stone in limestone, these particles should either be crushed to small pieces and scattered evenly or eliminated. In case of necessity, their content should be established by weighing and taken into consideration in the final calculation.

8.1.2.2 The gross sample obtained in this way is thoroughly mixed and reduced to the size required for a laboratory sample by suitable means.

8.2 Packing, Labelling and Destination of a Laboratory Sample

8.2.1 *Packing a Laboratory Sample*

The product constituting a laboratory sample should be packed into at least three containers in equal portions: sufficient for conducting all the scheduled tests.

8.2.1.1 The pouring in or putting in the product should be conducted in small portions. The portions should be placed successively one by one in each container, this action being repeated until the containers are filled up. When filled up, the containers should be secured in such a way as to ensure the identity of the sample.

8.2.2 *Inscription of the Packing of the Sample*

On each container a clear inscription should be placed including:

a) Name or number of the production plant or the supplier;
b) Designation of the product;
c) Unique identification of the consignment/date of production, number of wagon, specification number, etc;
d) Size of the consignment;
e) Name of the person who has taken the sample;
f) Date and place of sampling; and
g) Possibly also some other data included in the purchase contract.

8.2.3 *Labelling Sample Containers*

8.2.3.1 Label the containers suitably, immediately after a sample is obtained so that it withstands the usual rigours of storage and handling. Sufficient detail should be written on the label to completely identify the sample. The following information is frequently desired:

a) Date and time;
b) Name of the supplier;
c) Type and bulk containers from which sampled;
d) Name and/or registered number of the vessel, car or container;
e) Brand name, grade of material and code number;
f) Size of the consignment;
g) Name of the person drawing the sample; and
h) Reference symbol and necessary identification number.

8.2.4 *Destination of a Laboratory Sample*

The laboratory sample is designed for checking compliance of the product with the specification, carried out by the consignee or the consignor or by both for referee analysis. If a report of sampling is needed, it should be prepared in three copies.

8.2.5 At least four times as much reduced sample should be prepared as is required for one laboratory to perform a complete analysis. Retain one portion of the well-blended sample for the manufacturer or the seller;

one for the purchaser, one for the referee, and if necessary, one reserve to replace breakage or loss.

8.2.6 *Record of Sampling*

Immediately after taking the sample, a record should be written in triplicate. In the record the following data should be given:

a) Names of the supplier and the consignee;
b) Designation of the product;
c) Unique identification of the consignment, for example, date of production, number of wagon, specification number;
d) Size of the consignment;
e) Name of the person who has done the sampling;
f) Date and place of sampling;
g) A short description of the procedure of preparation of the samples, number and size of the increments, kind of instrument used in taking the increments, method of preparing the gross samples and the laboratory sample, size and method of packing and laboratory sample;
h) Possibly also some other data, for example, remarks on damaged packages in the consignment or accidental contamination of the product; and
j) Signature of the person who has done the sampling.

8.2.6.1 In the case of deviations from the principles of sampling established above or from the principles established by the standards for the products in question, this should be made clear in the record, and the justification of the fact that the method used in taking the sample should be given.

8.2.7 Samples that are to be stored over long periods, that may be affected by atmospheric exposure, or that may become seriously contaminated in contact with paper or cardboard should be packaged in widemouth screw-capped glass jars with caps and liners of suitable inert material.

8.2.8 For samples in which water content is important or composition is subject to change upon atmospheric exposure, plastic containers are generally unsuitable because of their permeability. In other cases, tight, leakproof paper sample envelopes or cardboard cartons with or without plastic liners or coatings, or even tin cans, may be used to hold sampler.

8.2.9 Where corrosion or atmospheric exposure causes problems it is usually better to use widemouth glass jars with suitable screw caps and liners.

8.2.10 In addition to precautions for storing samples mentioned, following precautions shall be observed.

8.2.10.1 Appropriate method of stacking for arranging packages shall be employed.

8.2.10.2 Care shall be taken of periodic inspection of various installations.

9 SAFETY IN SAMPLING

9.1 Introduction

In some cases, the act of taking a sample exposes the sampler to a risk of personal injury or may give rise to hazardous conditions endangering the safety of others. The recommendations in this standard are intended to assist those engaged in sampling, or directing the sampling and also those responsible for premises within which a sampling operation is performed, to ensure that sampling shall be a safe operation.

9.1.1 Users are reminded that there are a number of statutory regulations relating to the safety of personnel generally and to special industries and are advised to ensure that all statutory obligations are satisfied.

9.1.2 Attention is also directed to the existence of legislation regulating the carriage of dangerous goods and users of this standard should ensure compliance with these requirements and those imposed by the carriers.

9.2 General Recommendations

9.2.1 The following recommendations apply to all sampling operations whatever be the nature of the material being sampled. The operator shall have:

a) safe access to and from the place where the sample is taken; and
b) a safe working place with adequate light and ventilation.

9.2.1.1 There should be, if possible, two persons present, one engaged in sampling and the other to ensure his safety.

9.2.1.2 Sampling points in fixed installations should be arranged to satisfy these needs as well as any special ones arising from the nature of the material being sampled. Precautions shall be taken to ensure that the operator does not fall when sampling from the top of a tank or tank vehicle and against the collapse of stacked containers or solids in bulk. Adequate means shall be provided for extinguishing fire from any cause.

9.2.2 When the material being sampled is itself hazardous the following general recommendations shall apply.

9.2.2.1 The sampling operation shall be carried out in such a manner as not to prejudice the security of the bulk. This particularly applies to the sampling of fluids through cocks where the seizure of the cock in any open position could lead to the escape of large quantities of the fluid. It is recommended that the device used for such sampling be arranged so as to limit the total quantity drawn at any one time and to restrict the rate to flow to a safe limit. In the case of liquids it is desirable to assume that spillages will occur and to provide a drain to trap spilt liquids safely and to provide a permanent splash guard to protect the sampler. Both for liquids and gases whenever possible provision shall be made for isolating the sample point from the bulk or line by a valve near but not immediately adjacent to the sample point so that in the event of an accident the flow may be controlled from a safe place. In all cases it is part of the sampler's task to ensure satisfactory re-closure of all opened packages and sample points.

9.2.2.2 When it is necessary to purge or rinse a sample container with the material to be sampled and this material presents a hazard then appropriate facilities shall be provided for the disposal of material used in purging. Gases should be vented away from the vicinity of persons sampling or otherwise in the vicinity or from any hazard.

9.2.2.3 The size of the sample and the frequency of the sampling shall not be greater than are necessary for the examination proposed.

9.2.2.4 The sample in its container shall be carried in a suitable carrier designed and constructed to facilitate handling and to minimize the risk and consequences of breakage of the sample container.

9.2.2.5 The equipment including all tools and containers shall be compatible with the material being sampled and suitable for their purpose.

9.2.2.6 The sample or its container shall be marked to indicate the nature of the material and the risks associated with it.

9.2.2.7 The person taking the sample shall be made fully aware of the nature of the hazards involved and the precautions to be taken. He shall be instructed in the use of all appliances provided for his safety including fire extinguishers, protective goggles and clothing, etc. He shall be instructed to report to an appropriate supervisor before and after taking the sample and shall report, preferably to the same authority, any unusual happening or situation. Where toxic substances are being sampled he should be instructed that in the event of feeling unwell he should immediately report to his supervisor.

9.2.2.8 These general recommendations and the specific ones which follow should be used as a guide to the precautions necessary in preparing final samples.

9.2.2.9 It is stressed that those directing the activities of samplers must consider in detail the consequences of minor mishaps which may occur, such as spillage, failure of cocks, etc. The sampler must be given specific instructions covering both the normal situation and what he should do in the event of mishaps. Equally important are the specific instructions to be given to the safety observer present when toxic or dangerously corrosive materials are sampled

9.3 Specific Recommendations for Hazardous Materials

The physical or chemical properties of the material being handled may be such that they can have a direct physiological effect or be such that, for example, fire or explosion risks are present. The degree of risk is extremely variable and only general guidance is possible. A general classification of risks is listed below together with the appropriate precautions which are additional to those given in 9.2. It should be assumed in the absence of specific information to the contrary that any material being sampled is a hazardous one. Many materials present more than one hazard, for example, benzene is toxic, flammable and its vapour forms an explosive mixture with air. The following types of hazards may usually be encountered:

a) Explosive substances including unstable substances not used as explosives;
b) Oxidizing substances;
c) Flammable substances;
d) Toxic substances;
e) Corrosive and irritant substances;
f) Substances dangerous by virtue of their physical state, notably their pressure and temperature;
g) Radio-active material; and
h) Carcinogenic substances.

9.3.1 *Explosives and Unstable Substances*

9.3.1.1 *Example*

Unstable substances handled is water or other diluents, high strength hydrogen peroxide, ketone peroxides, peroxy organic acids and acetylene.

9.3.1.2 *Additional precautions*

a) Sample containers shall be closed so as to prevent loss of contents or evaporation of diligent, but the closure should provide for safe release of pressure.
b) Samples shall be protected from heat and shock and transported only in a carrier designed to retain the sample in the event of a leakage.
c) All spillages shall be reported for immediate action.
d) Naked lights and smoking shall be prohibited. Where appropriate, tools manufactured from

low-or reduced-spark generating materials shall be specified.

e) Protective goggles and protective clothing shall be worn.

9.3.2 *Oxidizing Substances*

9.3.2.1 *Example*

Liquid air and oxygen, oxidizing acids and their salts, hydrogen peroxide, etc.

The risk varies according to the substance and the combustible material it may contact and their degree of subdivision. It is important to remember that the sampler's clothing is almost certainly combustible.

9.3.2.2 *Additional precautions*

a) The vicinity of the sampling operation shall be free from combustible matter,

b) Adequate and appropriate fire extinguishers shall be available,

c) Sample carrier shall not include combustible padding material,

d) Naked lights and smoking shall be prohibited,

e) All spillages shall be reported and corrected as soon as possible, and

f) Protective goggles and protective clothing shall be worn.

9.3.3 *Flammable Substances*

9.3.3.1 Flammable gases, liquids and solids (typified by hydrogen, fuel oil and coal) including substances which although not normally regarded as being flammable themselves will give combustible products if exposed to moisture (alkali metal hydrides and calcium carbide are examples). Other substances will ignite spontaneously in contact with air, for example, white phosphorus, pyraphoric nickel and carbon. The risk varies with the substances, its temperature and its state of subdivision. It is generally higher with liquids than solids and is always high if the substance is volatile, yields flammable gases or is in a form which is readily dispersed in the air. Attention is particularly directed to the risk or explosion which is present in enclosed spaces where volatile fractions of the materials including residual traces of solvents, may accumulate and in places where dust rising out from combustible matter; such as flour, starch and coal will if dispersed yield an explosive mixture. Those responsible for the sampling must be acquainted with the flash and auto-ignition points, and the flammability limits of such substances.

9.3.3.2 *Additional precautions according to severity of risk*

a) The vicinity of the sampling operation shall be free of possible means or aids of ignition. Naked lights and smoking shall be prohibited. Where appropriate, tools manufactured from low-or reduced-spark generating materials shall be specified.

b) Precautions shall be taken to eliminate sparks due to the generation of static electricity by ensuring that the accumulation of static electrical charges cannot occur on electricity-insulated metal containers. Rubber tyred vehicles shall be earthed before operations commence. Metal sampling cans shall be earthed or bonded to the tank into which they are inserted. The flow of liquids can generate static electricity as can also the mixing of liquids and sufficient time should elapse after movement has stopped and before sampling is undertaken to ensure leakage to earth of the charge generated by movement. In fixed installations, sampling points shall be individually earthed if there is any possibility that they may become electrically insulated. There is also the possibility of the sample or his clothing being charged. To eliminate sparks between the sampler and earth, the sampler shall wear conductive footwear. Nylon overalls will often be highly charged in dry weather and cotton is to be preferred.

c) Adequate and appropriate fire extinguishers shall be available.

d) All spillages shall be reported and corrected as soon as possible; spilt flammable liquids should not be allowed to enter drains unless the system is known to be designed to take such fluids. Whenever possible time shall be allowed for the vapours to dissipate.

e) Protective goggles and protective clothing shall be worn. Such clothing shall not in itself be flammable.

f) Spontaneously flammable substance: shall be handled either under an inert liquid or in an inert atmosphere.

9.3.4 *Toxic Substances*

Poisons may enter the body by one or more of three: routes, namely:

a) Mouth and alimentary canal;

b) Respiratory system; and

c) Skin.

9.3.4.1 In acute poisoning, that is, from a single dose the effect may be immediate and associated with immediate discomfort and other symptoms, but in some cases there are delayed effects not appearing for some hours. Repeated low level doses of some substances are a health hazard because of accumulation of the poison itself in the body or by virtue of accumulated minor organic changes. When appropriate, persons exposed to this kind of risk shall be periodically examined by a doctor. Whenever toxic substances are handled or sampled those persons exposed to the risk shall be made aware of the risk, the symptoms of poisoning and the nature of any delayed effects and be instructed to seek immediate medical assistance in the event of their feeling unwell in any way. It is recommended that whenever the substance sampled may have a delayed effect, the sampler should be given dated card stating that he has handled the named substance and bearing the name and telephone number of a medical officer able to advise on the subject.

9.3.4.2 *Substances poisonous, if ingested via the mouth*

Under this heading are included solids and liquids of low vapour pressure since poisonous substances of appreciable vapour pressure will be regarded as presenting a major risk via the respiratory system. If the solid is so finely divided that airborne dust may arise then the risk should also be treated as for respiratory poison.

9.3.4.3 *Additional precautions according to severity of risk*

a) Smoking, tile use of snuff, eating and drinking in the vicinity of the poisonous substance shall be prohibited.

b) Adequate washing facilities shall be provided and be used by sampling personnel after securing sample containers and before leaving the site. Suitable facilities shall also be provided for adequate cleaning of all equipment after the samples are taken.

c) Sampling cocks for liquids shall be arranged so that splashing cannot occur and contained. Facilities shall be provided for isolating the sample point from the system by a bulb near to but not immediately adjacent to the sample point.

d) All sample containers and tools shall be clean and fit for use without rinsing with the product. Should rinsing be necessary or a sample line require initial purging, suitably marked containers shall be provided for the surplus liquid and format instructions issued directing the disposal of this surplus.

e) All spillages shall be reported immediately. As necessary the sampler shall wear suitable overalls so that in the event of these being contaminated he may change. Contaminated clothing shall not be sent to laundry or cleaners until a competent person, aware of the hazards involved, has taken the appropriate steps to remove the contamination.

9.3.4.4 *Respiratory poisons*

These include gases and volatile liquids and solids which are being handled in such a way that airborne sprays or dusts may arise. It is to be noted that in one sense all gases other than oxygen shall be regarded as presenting a health hazard in that very high concentrations will lower the oxygen content of the atmosphere being breathed. Even nitrogen, although forming the major part of the air one normally breathes; is potentially fatal in this sense. It follows that it is generally desirable that the area in which gases of any description under pressure are sampled should be well ventilated. When respiratory poisons present a health hazard personnel protection may be afforded either by providing a supply of fresh air to a suitable facemask or by providing a gas mask fitted with a canister containing the appropriate absorbent. For some gases, notably carbon monoxide, and whenever heavy concentrations may occur, only a supply of fresh air to a well fitting face mask shall be considered adequate. The absorbent canister protection should be used only when:

a) concentrations are low or the substance is relatively non-volatile and the absorbent is primarily acting as a filter for dust and droplet particles; or

b) the gas has an immediate and sustained physiological effect which will warn of its having penetrated past the absorbent, for example, ammonia, chlorine, etc, but not cyanide derivatives or hydrogen sulphide. Some individuals are virtually unable to detect the odour of hydrogen cyanide and awareness of hydrogen sulphide rapidly diminishes on continued exposure.

9.3.4.4.1 *Additional precautions*

a) The appropriate respiratory protection shall be provided for and used by all persons engaged in the operation.

b) The sampler shall be accompanied by a second person whose task is to ensure the safety of the sampler. During the sampling operation he will remain well clear of the sampling point and will observe the whole operation. He must be given specific instructions as to the action he will take in an emergency and such instructions shall always require that he first raises an alarm and does not attempt a rescue by himself.

c) Sampling cocks for liquids shall be arranged so that splashing cannot occur and spillages are contained. Facilities shall be provided for isolating the sample point from the system by a valve near to but not immediately adjacent to the sample point.

d) Sample containers shall be sealed before leaving the site.

e) All spillages shall be reported immediately. As necessary the sampler should wear suitable overalls so that in the event of these being contaminated he may change. Contaminated clothing shall not be sent to a laundry or cleaners until a competent person, aware of the hazards involved, has taken the appropriate steps to remove the contamination.

9.3.4.5 *Contact poisons*

Contact poisons enter the system through the skin and are to be distinguished from corrosive poisons covered in **9.3.5**. In general, corrosive substances are those which cause immediate damage to the tissues they contact and are noxious solely because of this damage. Contact poisons including carcinogenic substances on the other hand are often absorbed through the skin into subcutaneous tissues without necessarily causing immediate surface damage or sensation. There may or may not be a subsequent skin reaction at the site of entry. A few substances, for example, hydrofluoric acid, are both corrosive and in this sense contact poisons, but in general the contact poisons are more dangerous than corrosive poisons since entry into the system is not immediately apparent to the victim. The vapours of substances in this category should be assumed to be able to enter the body via the skin as readily as the liquid or solid itself and will in any case be respiratory poisons. All these substances are poisonous if taken in by the mouth. It is recommended that unnecessary skin contact with any organic liquids should be avoided and that regardless of the nature of the chemicals being handled a sampler shall wash his hands free from contamination.

a) *Additional precautions according to severity* — The appropriate protective clothing should be provided and worn. According to the severity of the risk this will be:

    1) full protective impervious outerclothing and a respirator;

    2) impervious apron, gloves, boots and respiratory or face mask; or

    3) face mask and gloves.

    Attention is directed to the risk of unsuitable footwear acting as a trap for splashes.

b) Adequate washing facilities shall be provided including a warm water shower for preference or as a second alternative a cold water shower. Before commencing work, the sampler should test that the shower is in working order. Absorbent fibre should be available to wipe off any oily material before washing.

c) Contaminated clothing shall be immediately removed.

d) The sampler shall be accompanied by a second person whose task is to ensure the safety of the sampler. During the sampling operation he will remain well clear of the sampling point and will observe the whole operation. He must be given specific instructions as to the action he will take in an emergency and such instructions should always require that he first raises an alarm and does not attempt rescue by himself.

e) Sampling cocks for liquids should be arranged so that splashing cannot occur and any spillage is contained. Facilities shall be provided for isolating the sample point from the system by a valve near to but not immediately adjacent to the sample point.

f) All sample containers and sampling apparatus shall be clean and fit for use without rinsing with the product. Should rinsing be necessary or a sample line require initial purging, suitably marked containers shall be provided for the surplus liquid and formal instructions issued directing the disposal of this surplus.

9.3.5 *Corrosive Substances and Irritants*

The risks involved under this heading include the well known rapid effects of strong acids and alkalis, but attention is directed to the general undesirability of unnecessary exposure of the skin to any chemical. Comparatively harmless substances, such as soda ash can cause dermatitis; for others sensitisation may occur. The precautions to be taken are similar to those advised for contact poisons, but particular emphasis is placed on the use of safety goggles for eye protection. The corrosive action of strong acids and alkalis is such that immediate action to dilute them is absolutely essential and it is imperative that a drench shower or a plunge bath be available at the sampling point. All washing and drenching facilities must be shown to be in working order before commencing sampling. Additional precautions, as appropriate, should be taken as described in **9.3.4.3**.

9.3.6 *Substances Dangerous by Virtue of their Physical State*

These include substances at extreme temperatures and high pressures. In general, extremely cold and hot substances present similar hazards to those of corrosive substances except that damage is virtually instantaneous and drench showers are of doubtful value.

9.3.6.1 *Additional precautions for substances dangerous by virtue of temperature*

a) Eye protection against splashes is essential.
b) For very hot substances shielding of the face and neck against radiant heat will be needed and additional protection of the eyes against radiation is needed.
c) The hands shall be protected against, splashes by gloves or gauntlets that will not readily absorb the material being handled.
d) Aprons may be needed. Footwear shall be stout and afford adequate protection and, in particular, should not fit so as to trap splashed material.

9.3.6.2 *Additional precautions for substances dangerous by virtue of pressure*

The sampling of fluids under pressure may be achieved either at atmospheric pressure or at system pressure.

a) The sampling arrangement shall include suitable devices for restricting the rate of flow at the exit from the high pressure system to a safe level and the exit itself shall be of such a bore that the velocity of escape of the fluid is not capable of causing injury.
b) When the sample is to be withdrawn at system pressure the sample container shall be inspected at frequent intervals by a competent person who shall certify its suitability for use up to a declared pressure which shall be marked on the container. The container shall be used for this purpose only. The fittings for attaching the container to the sample point shall be adequate for the system; the sampler shall use the correct tools for attaching the container to the point and shall check the security of the union before proceeding to take the sample.
c) Sample containers shall be only filled with a liquid so as to provide adequate ullage, which in all cases shall not be less than 5 percent at the maximum temperature likely to be encountered.

9.3.7 *Radio-Active Substances*

9.3.7.1 *Introduction*

Various statutory regulations and recommendations detail the principles and procedures for restricting exposure to ionizing radiation and for preventing or limiting inhalation and ingestion of radioactive substances. Under normal circumstances, all radioactive materials are handled only by suitably trained personnel in specialized establishments where the necessary equipment, containment facilities and advice from qualified health physicists are available. Under any other circumstances:

> NO APPROACH TO, OR HANDLING OF, RADIOACTIVE MATERIAL SHOULD TAKE PLACE UNTIL EXPERT AND COMPREHENSIVE ADVICE HAS BEEN OBTAINED.

9.3.7.2 *General recommendations*

It is essential that sampling operations may be carried out only by adequately trained personnel. In this regard, reference should be made to the Department of Atomic Energy (Division of Radiological Protection, BARC).

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Inorganic Chemicals and Photographic Materials Sectional Committee, CHD 1

Organization	*Representative(s)*
Central Salt and Marine Chemicals Research Institute (CSMCRI), Bhavnagar	Dr P. K. Ghosh (***Chairman***) Dr. R. S. Shukla (***Alternate***)
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Ballarpur Industries Limited, Karnataka	Dr V. V. Savant Dr R. S. Ramachandra (***Alternate***)
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Geological Survey of India, Kolkata	Dr D. K. Das Dr Subhash Chandra (***Alternate***)
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Member Secretary
Shri P. Ghosh
Director (CHD), BIS

Analytical Method Validation for Inorganic Chemicals Subcommittee, CHD 1 : 1

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